[0055] FIGS. **1**(*a*) through **1**(*f*) show examples trench or via **2** filling within a dielectric substrate **1**. The dielectric substrate **1** may be first metallized with an electric conductive material on the surfaces adjacent to metal **3**, not shown. FIG. **1**(*a*) shows subconformal filling of a metal **3** in trench or via **2** in a planar region **4** of the dielectric substrate. The subconformal filling has a greater deposition rate at pattern features such as at the top edges **5** of trench or via **2**. The subconformal growth shown in FIG. **1**(*a*) may lead to voids **6** as shown in FIG. **1**(*b*). As the electrodeposition continues, the metal growth at top edges **5** continues until the metal **3** meets at point **8**, forming void **6** therein as shown in FIG. **1**(*b*). FIG. **1**(*c*) shows an example of conformal growth wherein metal **3** grows at a consistent rate on the planar region **4** of the dielectric substrate, within trench or via **2**, and at the top edges **5** of trench or via **2**. Conformal growth as shown in FIG. **1**(*c*) may form a seam **8** as shown in FIG. **1**(*d*). Seam **8** may be formed with the continued growth of metal **3** inward from the sidewalls **7** of trench or via **2**. FIG. **1**(*e*) shows an example of superconformal super-filling wherein metal **3** forms a "V-notch" **9** centrally oriented within trench or via **2**. This is also referred to as bottom-up filling and may produce a substantially uniform filling of trench or via **2** with metal **3** as shown in FIG. **1**(*f*). In bottom up filling, metal deposition occurs preferentially in recessed surface features, such as patterned trenches and vias **2**, thereby resulting in void-free filling, as shown in FIG. **1**(*f*). On freshly immersed substrates, trench filling may be characterized by an initial period of uniform growth as shown in FIG. **1**(*e*) followed by the development of "v-notch" **9** geometry which may be associated with transient depletion of an additive such as MBIS or PEI within the recessed feature, trench or via **2**. The finest submicrometer features may be filled with only minimal deposition on the neighboring free surfaces **10**, as shown in FIG. **1**(*e*).

EXAMPLES

[0056] The following examples are included to demonstrate embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples that follow represent techniques discovered by the inventors to function in the practice of the invention, and thus can be considered to constitute selected modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments that are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention.

Example 1

[0057] Void-free Ni deposition was shown onto a dielectric substrate. Nickel was eletrodeposited from an electrolytic bath comprising of 1.0 mol/L $NiSO_4.6H_2O$, 0.2 mol/L $NiCl_2.6H_2O$, and 0.5 mol/L H_3BO_3, with pH 2.5 as a base electrolyte. Separate electrolytic solutions were prepared by adding different concentrations of MBIS, from 0 to 400 μmol/L, to the base electrolyte. The sodium salt dihydrate of MBIS, $C_7H_5N_2NaO_3S_2.2H_2O$, manufactured by Aldrich, was used. Both planar and patterned Cu seeded Si wafers were used as working electrodes for electrochemical analyses and film growth. The specimens were prepared by physical vapor deposition of a thin Ti adhesion layer followed by 100 nm of Cu. The trenches including the barrier layer were 670 nm deep, and the widths varied from 590 to 90 nm at the bottom and from 720 to 120 nm at the top, corresponding to sloping sidewall angle that ranged from 6 to 2 degrees from vertical. On the basis of the average width, the features range from 6.4 to 1.0 in aspect ratio (height divided by average width). In this example, features are specified by the respective bottom width.

[0058] The working electrodes were sealed with a Teflon holder having an exposed area of 1.28 cm , facing up to the electrolyte in order to facilitate removal of any hydrogen gas associated with deposition. The Pt counter electrode was formed as a circular wire of similar radius to, and suspended above, the working electrode. A saturated calomel reference electrode (SCE) was firmly positioned with respect to the working and counter electrode in a Teflon jig, thereby fixing the electrical distribution and resulting internal-resistance iR effects between different solutions. For feature filling examples, the trench and via patterned wafer specimens were immersed at a preset growth potential. Field-emission scanning electron microscopy (FESEM) was used to examine cross sectional profiles of the trench filling. Transmission electron microscopy (TEM) was used to more closely examine the microstructure of an array of Ni-filled trenches. The specimen was prepared by focused ion-beam FIB sectioning. X-ray diffraction (XRD) and atomic force microscopy (AFM) were used to examine the crystallographic texture and the surface morphology of the Ni films grown on planar substrates. The magnetic properties of planar Ni and Ni—Fe films were examined by vibrating sample magnetometer (VSM) and the saturated magnetic moment, coercivity and squareness were evaluated as a function of MBIS concentration. Specimens were prepared by cleaving to obtain square-shaped samples. The specimen area and thickness were measured by an optical scanner and FESEM, respectively.

[0059] Ni deposition from the additive-free electrolytic bath was examined by cyclic voltammetry (CV). The onset of metal deposition was evident near 0.7 V, followed by a rapid increase in the current with the applied potential that tends toward a linear response at higher current densities.

[0060] A series of samples were feature filled as a function of MBIS concentration. Ni was deposited at −0.925 V SCE (i.e., voltage defined relative to saturated calumel reference electrode, SCE) for 3 min, and then the specimens were cross sectioned for FESEM analysis. Images of trench arrays with four different dimensions are shown in FIG. **2** wherein trench widths of 520 nm are shown in column (**1**), trench widths of 260 nm are shown in column (**2**), trench widths of 150 nm are shown in column (**3**), and trench widths of 90 nm are shown in column (**4**). The trench widths are reported as the dimension of the bottom of the trench and these different dimensions offer a representative sample of filling behavior. The concentrations of MBIS were 0 μmol/L, shown in row (a), 50 μmol/L, shown in row (b), 80 μmol/L, shown in row (c), 100 μmol/L, shown in row (d), and 150 μmol/L, shown in row (e). The dark contrast **103** at the bottom **118** of the trench **102** and on the free surface **104** between the trenches is associated with the copper seed layer. In the absence of MBIS, a large seam **106** is apparent in the narrowest trenches, shown in FIG. **2**, column (**4**), while voids are sometimes evident at the top of all but the widest features, as in row (a) of FIG. **2**. The upper surface **110** of the deposition on free surface **104** is also notably rough relative to the dimension of the trenches **102** and overburden **112**. Thus, conformal deposition coupled